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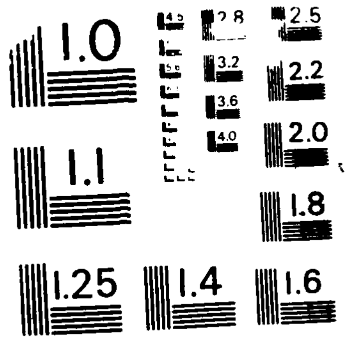

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II. OBSERVATIONS ON PARTICLE LOSS (U)

by

J.R. Coleman

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ABSTRACT

As part of a program of filter testing, monodisperse aerosols ranging in size from 0.01 to 0.30 µm were prepared from seventeen materials. With particles of a few hundredths of a micron diameter, losses in the test system lines were frequently much larger than expected, and varied greatly from one material to another. There was no obvious correlation between the magnitude of the effect and the chemical nature of the aerosol. These abnormal losses were greatly reduced or eliminated when the aerosol was passed through a neutralizer, which removed most of the particle charge in this size range. A more systematic study of the phenomenon should be made, with a system designed expressly for the purpose. Canada

RÉSUMÉ

Dans le cadre d'un programme d'essai de filtres, des aérosols monodispersés, dans la plage de diametres de 0,01 à 0,30 µm, ont été préparés à partir de dix-sept substances. Avec des particules de quelques centièmes de micron de diamètre, les pertes dans les canalisations du système d'essai sont souvent beaucoup plus élevées que prévu, et varient beaucoup d'une substance à l'autre. Il n'y a aucune corrélation évidente entre la grandeur de l'effet et la composition chimique de l'aérosol. Ces pertes anormales sont grandement réduites, voire éliminées, lorsque l'aérosol est passé dans un neutralisant qui élimine la plus grande partie de la charge des particules dans cette plage de diamètres. Une étude plus systématique du phénomène au moyen d'un système conçu expressément pour cette fin est recommandée.

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1.0 INTRODUCTION

The filter test system FTS 400 has been employed in several investigations at DREO (1,2). It can generate aerosol challenges at discrete, narrowly defined sizes selectable between 0.01 and 0.30 µm. In Part I of this Note (3) it was reported that in the upper end of this range, above 0.10 µm, test results with a standard filter are nearly independent of the challenge material, but that at small sizes penetration varied greatly with aerosols of different chemical nature.

The aerosol particles produced in the original FTS 400 were charged unipositively at all sizes. It was found of advantage to insert a radioactive neutralizer in the line conveying aerosol, to reduce particle charge to the Boltzmann equilibrium distribution. Theoretical calculations of the Boltzmann distribution as a function of particle size have been made; a few examples from a recent publication (4) are listed in Table I.

TABLE I

Percent of Particles Carrying n Elementary Charge Units

D, µm	n=-4	-3	-2	-1	0	+1	+2	+3	+4
0.01				0.34	99.32	0.34			
0.02				5.23	89.53	5.23			
0.04			0.23	16.22	67.10	16.22	0.23		
0.06		0.01	1.25	21.30	54.88	21.30	1.25	0.01	
0.08		0.08	2.78	23.37	47.53	23.37	2.78	0.08	
0.10		0.26	4.39	24.09	42.52	24.09	4.39	0.26	
0.20	0.32	2.33	9.66	22.63	30.06	22.63	9.66	2.33	0.32

In contrast to the original uniform +1 charge at all sizes, the neutralizer removes all or most of the charge on the smallest particles; at larger sizes there is a rearrangement, with the appearance of a significant fraction of uncharged, and small fractions of multiply charged particles.

Working with particles in the upper size range, the presence or absence of the neutralizer had little effect on filter penetration of standard filters (2); but below 0.1 µm it affected both penetration

results, and, as is shown in this note, aerosol losses along the transport lines of the FTS, in ways that differed with different challenge materials.

Percent penetration is defined as the ratio of downstream/upstream concentrations × 100. In the design of such test systems, it is assumed that aerosol losses along the upstream and downstream sampling paths are small and self-cancelling, and so do not affect the final result. In this apparatus the upstream and downstream lines, which are described below, differ in geometry and construction material. Preliminary work with NaCl and DOP challenges (2) when no filter was under test, so that both paths were unimpeded, showed that for +1 charged particles NaCl loss was appreciably greater for the upstream than for the downstream path. No such difference was observed using DOP. The effect with NaCl aerosols became insignificant at sizes > 0.05 μm; and introduction of a neutralizer in the aerosol line before the test chamber removed it.

The filter test work of Part I which used a number of aerosol materials afforded a chance to look more extensively at line loss effects, and these form the subject of this note. When filter penetration measurements were completed for each challenge in turn, the filter was removed and aerosol behaviour in the line was studied. These results apply of course only to the actual flow paths found in a particular apparatus, the FTS 400, in work incidental to filter testing.

2.0 EXPERIMENTAL

The FTS 400 and its operating procedures have already been described (1).

2.1 AEROSOL MATERIALS

The same sixteen chemicals in the same stock solutions were used here and in the work described in Part I of this note. They are listed in Table II. Dibutyl sebacate, which with its congeners has been proposed as an alternative to DOP in filter testing, was also employed. For this material two stock solutions were prepared in isopropanol, of the same concentration as the DOP solutions.

TABLE II

Apparent Percent Penetration (Downstream/Upstream) Concentration × 100, with Test Compartment Empty (a) with Neutralizer; (b) Without Neutralizer

Aerosol Material	Diameter μm						
	0.01	0.02	0.05	0.10	0.15	0.20	0.30
	High Penetration						
Glycerol							
(a)	98	100	103	109	104	107	103
(b)	247*	411*	366	178	130	110	102
Phthalic Anhydride							
(a)	93	100	101	99	102	100	100
(b)	680*	309*	127	100	102	103	99
Dibutyl Sebacate							
(a)	-	-	-	-	-	-	-
(b)	890*	365*	111	100	101	100	99
Anthracene							
(a)	74	93	99	118	117	107	99
(b)	412*	256	121	109	113	99	99
Urea							
(a)	88	95	100	102	100	98	97
(b)	469*	519*	207	103	103	100	91
	Intermediate Penetration						
NaCl							
(a)	96	99	101	101	100	101	98
(b)	184	128	110	104	100	99	99
Glycine							
(a)	95	108	103	99	113	98	100
(b)	102	145	108	103	108	100	100
NaK Tartrate							
(a)	90	105	106	104	102	101	100
(b)	99	139	115	103	102	101	99

* Highly variable, but always hundreds of percent. The values quoted give only a representation of the magnitude. Results for glycerol at 0.01 μm (all values) are: 247, 572, 177, 419, and at 0.02 μm: 411, 466, 230, 231.

TABLE II (Cont'd)

Aerosol Material	Diameter μm						
	0.01	0.02	0.05	0.10	0.15	0.20	0.30
Oleic Acid							
(a)	82	98	107	103	101	96	98
(b)	83	166	110	102	103	100	96
PEG 400							
(a)	99	100	104	101	102	98	99
(b)	130	179	106	101	101	101	99
Paraffin Oil							
(a)	93	92	103	101	103	99	98
(b)	98	188	115	100	100	101	99
Silicone Oil							
(a)	60	102	102	102	101	101	100
(b)	51	175	110	101	101	101	99
Triethylene Glycol							
(a)	90	98	102	101	103	102	101
(b)	190	140	114	110	104	103	101
Oxamide							
(a)	68	101	102	101	102	102	99
(b)	92	143	105	101	101	98	93
	Low Penetration						
Dextrose							
(a)	96	105	105	102	103	102	99
(b)	99	121	107	102	103	101	103
DOP							
(a)	88	95	100	100	101	108	100
(b)	45	122	103	101	102	101	102
Citric Acid							
(a)	94	102	103	103	101	101	101
(b)	100	116	107	102	101	100	100

In addition, a few experiments were conducted using stock solutions with two materials in the same solvent, so that individual droplets or particles of the resulting aerosol were inhomogeneous. These will be described below.

2.2 FLOW PATHS

Figure 1 depicts the transport lines in the system for generating submicron particles.

From the electrostatic classifier the aerosol passes to the dilution manifold via tube A. This is $^3/_{16}$" i.d. steel tubing of total length about 24", in which is inserted a neutralizer (TSI 3077). In certain work this was replaced by a $^5/_{16}$" i.d. x 34" long glass tube with an expansion bulb (~ 1" diameter) at the end nearest the classifier. The tube was coated on the inside with conductive silver paint to eliminate static charge buildup, so that it should be equivalent in this respect to the metal tube. It had the advantage that a radioactive neutralizer could be applied at will to the expansion bulb and removed. From the all metal dilution manifold aerosol passes via tygon tube B (~ $^3/_4$" i.d. x 12" long) to the test chamber. The upstream sampling tube C is $^9/_{16}$" i.d. x 18" long tygon conducting aerosol to a common chamber from which the CNC samples. The downstream path D is all metal, the 4" i.d. sample chuck necking down via $^1/_2$" i.d. x 3", then $^5/_{16}$" x 6" tubing to the same common chamber. Tygon does not readily acquire static charge, and is suitable for aerosol handling instrumentation. In the submicron range inertial effects connected with changes in flow direction are insignificant, and it seemed reasonable to suppose that losses should be small throughout the system described above.

2.3 UPSTREAM-DOWNSTREAM DIFFERENTIAL LOSSES

If the assumption that there are only small line losses is correct, then when a run is conducted with no filter in place, the measured upstream and downstream concentrations should be nearly equal, and an apparent "penetration" of 100% or close to it should be obtained in every case.

In a test of this assumption, such apparent penetrations were measured using all the challenges referred to above. The appropriate

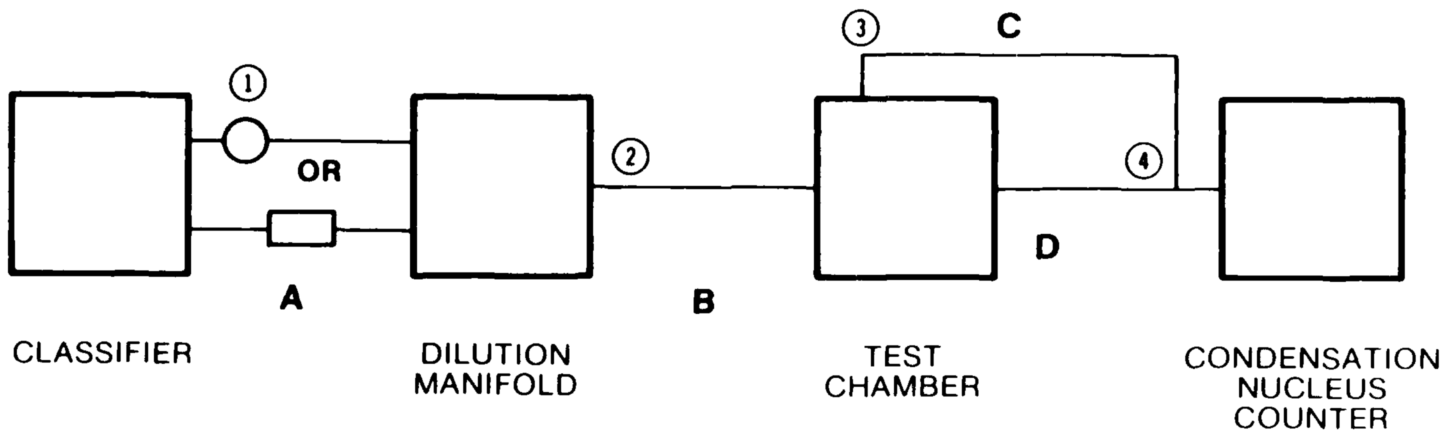

Figure 1: Schematic of flow paths in submicron aerosol generating section of FTS 400. Symbols are explained in text. The classifier and the dilution manifold are joined by a silvered glass tube with expansion (upper), or a steel tube with TSI neutralizer inserted (lower).

filter test program was run, with seven sizes between 0.01 and 0.31 μm, and with the sample holder empty. The flow rate was 16 LPM as before. To determine the influence of particle charge, the line with the TSI neutralizer was employed first (section A, Figure 1) and then replaced by the silvered glass tube.

2.4 AEROSOL LOSSES IN LINE

Evidence from earlier work (2) indicated that removal of charge from some aerosols reduced losses in passage through the system. In such cases the application of a neutralizer to the line at points progressively closer to the aerosol source should increase the concentrations measured at the CNC. The 100 millicurie Sr^{90} source described earlier (2) was used here since, not being enclosed, it could be applied to any non-metallic (glass or plastic) part of the line. This produced a β-particle flux sufficient to establish through the tubing walls charge equilibrium in the aerosol passing through. Shielding was required when the source was deployed about the FTS.

The source was applied at several places in the line, numbered as in Figure 1.

1. On the silvered glass bulb adjacent to the classifier (the bulb increased aerosol residence time in the β-particle beam sufficiently to ensure attainment of charge equilibrium).

2. On the tygon inlet tube adjacent to the dilution manifold.

3. On the tygon upstream sampling tube adjacent to the test chamber.

4. At the other extremity of the sampling tube.

In these experiments aerosol of the size and material desired was generated. During sampling by the upstream path, the concentration was allowed to reach a steady value close to equilibrium, as judged by visual observation of the continuously updated readout of the CNC. This reading was noted and the Sr^{90} source was then applied at the four positions in succession and the transitory change in the CNC reading noted for each position. This change began within 2-3 seconds and reached a new plateau in about 20-30 seconds. These readings are necessarily only approximate: if final equilibrium is reached, the FTS program will switch to downstream sampling, changing the path and disrupting the experiment. From the trend in CNC readings as the neutralizer is applied at points successively farther from the classifier, one can get a rough measure of aerosol disappearance in the line. These results are comparative, as between different aerosols, and not absolute.

3.0 RESULTS

3.1 UPSTREAM-DOWNSTREAM DIFFERENTIAL LOSSES

Results for the sixteen materials used in Part I, and for dibutyl sebacate, are presented in Table II, as apparent percent penetration at seven sizes, in presence and absence of the TSI neutralizer They are grouped roughly into materials of large, intermediate and little or no loss, on the basis of values at a few hundredths of a micron.

With most charged aerosols there is a measurably greater loss of aerosol by upstream than by downstream paths at the lower sizes, reflected in apparent penetrations of well over 100%. This difference rapidly diminishes at larger sizes and is negligible in most cases at 0.05 µm and above; glycerol is exceptional, with perceptible differential losses at 0.15 µm.

Insertion of the neutralizer eliminates the effect, the two measured concentrations becoming equal within experimental error over the entire size range (aside from two anomalous results, anthracene and silicone oil at one size, 0.01 µm). At the largest sizes, 0.2-0.3 µm, all results are close to 100%, implying that line losses become negligible by both paths and that charge state is immaterial; inertial forces are dominant.

From Table II no correlation can be seen between the extent of the differential loss and any chemical or physical property of the aerosol material; this is apparent from the position in the table of the diamides urea and oxamide; the diesters DOP and dibutyl sebacate; the polyols glycerol, PEG 400 and triethylene glycol; citric and oleic acids, glycine and phthalic anhydride. Liquid and solid materials are found in all three groups.

The original supposition was that the tygon section of the upstream sampling path would provide a greater opportunity for static charge buildup, and that greater losses would occur here. The all metal downstream path was taken implicitly as the reference, and upstream losses measured against it; and in fact upstream losses are generally greater, from the apparent penetration results; any significant divergences from 100% are in the positive direction. However, static problems are generally not important with tygon; furthermore, in preliminary work using NaCl (2), which displays differential losses in a moderate degree, it was found that rendering the tygon tube completely conductive with silver paint did not diminish the effect. Indeed, the upstream path is wider and has fewer

constrictions than the downstream, and it is difficult to understand why aerosol should deposit more readily along it.

It was observed also that "penetrations" in the vicinity of 100% are reasonably reproducible (within 5-10%); while the extremely high values for say 0.01 μm urea or phthalic anhydride fluctuate widely from run to run, though always remaining in the region of hundreds of percent. These fluctuations turned out to be due mainly to highly erratic upstream concentrations, the downstream values remaining nearly constant. The presence of static charge would be the most likely explanation, were it not apparently ruled out by the observations above.

3.2 BEHAVIOUR OF AEROSOLS FROM MIXED MATERIALS

It was seen that upstream sample losses were slight with citric acid and DOP. Aerosols were prepared from these materials with a mixture of a small proportion of materials for which the effect was pronounced, citric acid +5% w/w urea, citric acid +10% phthalic anhydride, DOP +10% glycerol, DOP +10% dibutyl sebacate. Stock solutions of these mixtures were prepared as before, with only one modification: phthalic anhydride is very insoluble in water, and citric acid + phthalic anhydride solutions were prepared in ethanol.

Experiments were carried out as before with these mixed aerosols. Table III lists apparent penetration results obtained with these mixtures, and Figures 2 and 3 show results graphically for both the mixtures and the base constituents. Comparing the two figures it is seen that, with composite particles of citric acid and the second material, the same differential loss occurs, but in attenuated form. With DOP mixtures, penetrations are barely if at all distinguishable from results with DOP alone.

Aerosols based on citric acid with urea or phthalic anhydride are probably aggregates of microcrystals, with the additive responsible for differential loss occupying some fraction of the surface. DOP, glycerol and dibutyl sebacate (DBS) are liquids. DOP is only slightly soluble in glycerol, but miscible with DBS. Spherical droplets are presumably formed, with the DBS dissolved in DOP, the glycerol perhaps in the form of droplets encapsulated in DOP, but in either case not much in evidence on the surface. This is conjectural, but in any attempt to explain the differences in behaviour it is useful to consider among other things the differing aspects the aerosol droplets and particles present on the microscopic level. From the incomplete evidence it appears that the presence of the material on the surface as a pure chemical is necessary for display of the abnormal losses.

TABLE III

Apparent Percent Penetration (Downstream/Upstream) Concentration × 100, With Test Compartment Empty, Without Neutralizer

Aerosol Material	Diameter µm						
	0.01	0.02	0.05	0.10	0.15	0.20	0.30
Citric Acid + 5% Urea	148	183	112	101	101	102	100
Citric Acid + 10% Phthalic Anhydride	298	216	116	92	105	101	100
DOP + 10% Glycerol	61	129	106	101	104	100	100
DOP + 10% Dibutyl Sebacate	51	151	106	102	100	101	100

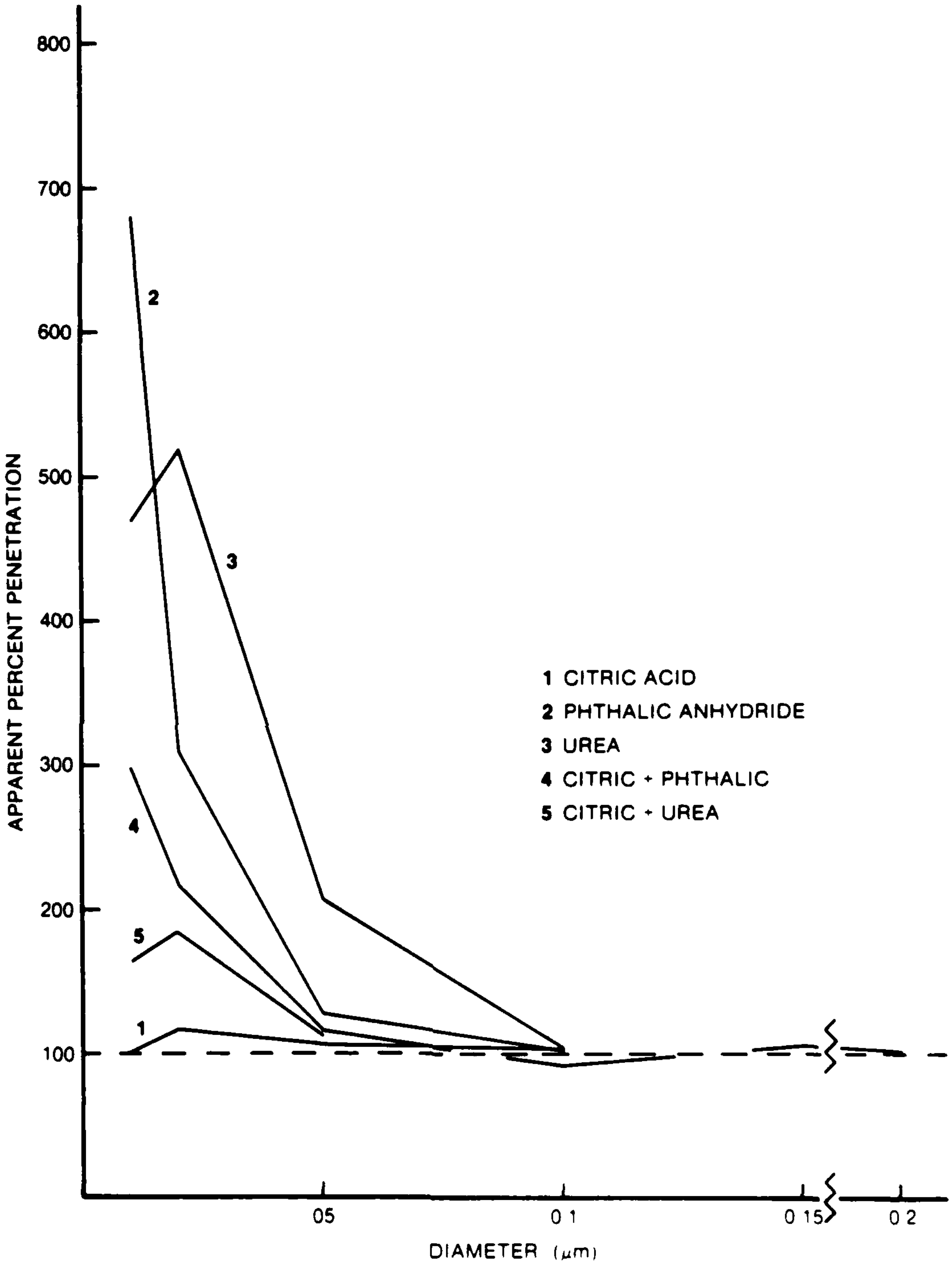

Figure 2: Apparent penetration, test chamber empty, for aerosols of citric acid, urea, phthalic anhydride, and blends of these.

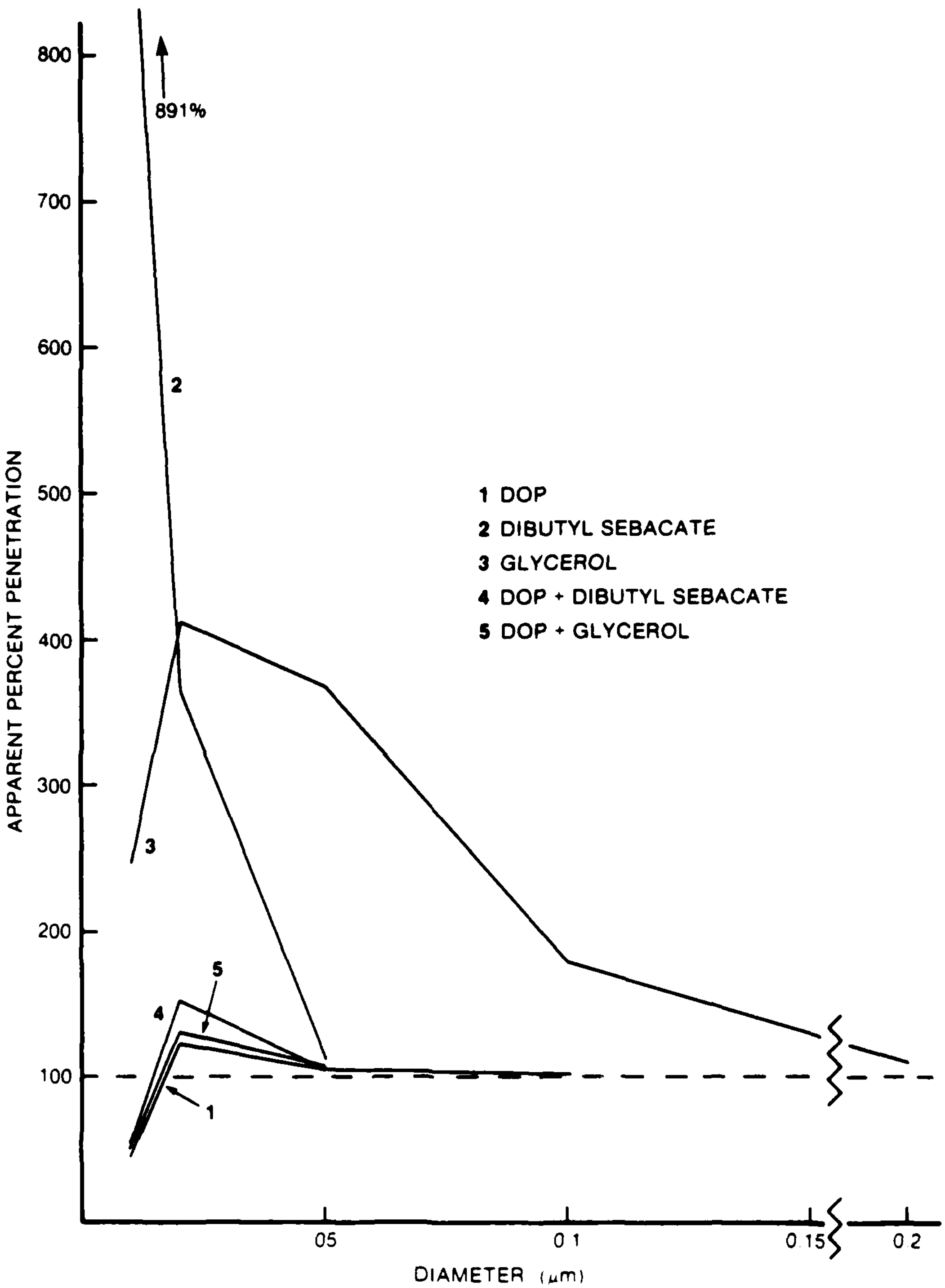

Figure 3: Apparent penetration, test chamber empty, for aerosols of DOP, glycerol, dibutyl sebacate, and blends of these.

3.3 IN-LINE LOSSES

Of the sixteen materials, data are presented for five - NaCl, glycerol, DOP, citric acid and urea - which in the previous section were found to display upstream losses at levels from none to great. Four particles sizes were used, in the range 0.01-0.10 μm. In Table IV, column 3 presents measured upstream concentration data in the absence of the neutralizer, and the succeeding four columns the corresponding values when the neutralizer is applied, first, immediately next to the classifier where aerosol is generated, and then at the three stages farther downstream shown in Figure 1.

In brief these results substantiate the observations made already. At one extreme of behaviour, with glycerol and urea, application of the neutralizer increases upstream concentrations substantially (by factors of about 12 and 7 at the 0.01 μm size) with a considerable dropping back in concentration at the succeeding positions. The effect again becomes smaller at increasing size. This is consistent with the charged aerosol suffering substantial in-line losses.

At the other extreme are citric acid and DOP, in which differential losses were small or negligible. Both the increase in CNC readings on applying the neutralizer at position 1, and the subsequent fall off as the point of application is moved downstream are slight (citric acid) or nil (DOP).

4.0 DISCUSSION

Both types of experiment described here measure the disappearance of aerosol particles passing through the system, presumably by capture on the walls. The losses vary greatly and with some materials are considerably greater than anticipated. The flow rate is 16 LPM and the volume of the system including test chamber about one litre, so that the time of passage is a few seconds and there is no opportunity for more than a small fraction of the aerosol to approach the walls of the transport lines.

The coagulation of aerosols has been discussed by Green and Lane (5). This is ruled out as an explanation of line losses on two accounts.

TABLE IV

Measured Aerosol Upstream Concentrations
Neutralizer Applied at Several Positions

Aerosol Material	Neutralizer Position					
	Size μm	None	1	2	3	4
Glycerol	0.01	6.0×10^1	7.0×10^2	6.2×10^2	5.5×10^2	4.5×10^2
	0.02	2.5×10^3	1.0×10^4	9.5×10^3	8.4×10^3	8.7×10^3
	0.05	5.7×10^3	2.0×10^4	1.9×10^4	1.8×10^4	1.7×10^4
	0.10	2.1×10^5	3.2×10^5	3.1×10^5	3.1×10^5	3.1×10^5
Urea	0.01	1.0×10^2	7.4×10^2	6.6×10^2	6.6×10^2	3.5×10^2
	0.02	3.8×10^2	5.0×10^3	3.6×10^3	3.8×10^3	2.5×10^3
	0.05	3.1×10^4	5.2×10^4	5.1×10^4	4.8×10^4	4.3×10^4
	0.10	3.3×10^5	3.2×10^5	3.3×10^5	3.4×10^5	3.3×10^5
NaCl	0.01	1.5×10^2	7.5×10^2	6.0×10^2	6.2×10^2	5.1×10^2
	0.02	3.3×10^4	5.9×10^4	5.7×10^4	5.2×10^4	5.0×10^4
	0.05	2.1×10^5	2.4×10^5	2.4×10^5	2.3×10^5	2.3×10^5
	0.10	3.9×10^5	3.9×10^5	3.8×10^5	3.8×10^5	3.9×10^5
DOP	0.01	3.0×10^1	3.1×10^1	3.1×10^1	3.0×10^1	2.9×10^1
	0.02	3.5×10^2	3.9×10^2	3.5×10^2	3.5×10^2	3.0×10^2
	0.05	2.7×10^4	3.1×10^4	3.0×10^4	3.0×10^4	2.9×10^4
	0.10	7.9×10^4	8.0×10^4	8.0×10^4	7.8×10^4	8.0×10^4
Citric Acid	0.01	5.2×10^1	1.4×10^2	1.3×10^2	1.1×10^2	7.5×10^1
	0.02	5.5×10^3	1.0×10^4	9.3×10^3	9.0×10^3	7.5×10^3
	0.05	1.6×10^5	1.9×10^5	1.9×10^5	1.8×10^5	1.8×10^5
	0.10	5.3×10^5	5.7×10^5	5.6×10^5	5.5×10^5	5.5×10^5

a) The effect is seen only with uniformly +1 charged particles, and mutual repulsion will hinder and not promote coagulation in the air-stream.

b) Only particles of the smallest size are affected. Upstream and downstream concentrations were recorded for each size in all the runs; the range of values is:
0.01 μm: $10^1 - 2 \times 10^3/cm^3$ 0.02 μm: $10^2 - 4 \times 10^4/cm^3$
0.05 μm: $2 \times 10^4 - 3 \times 10^5/cm^3$.

All of these, especially the values at 0.01 and 0.02 μm, are well below the range at which any appreciable coagulation can occur in the several seconds transit time.

There has been some speculation about the role of particle shape in fibrous filter action (6). Invoking shape factors to explain these losses encounters the difficulty that large differences in behaviour were observed here among aerosols of liquid materials, all of them presumably spherical, so that this could not in any case provide the complete explanation.

If an aerosol has any appreciable volatility, loss of the smallest particles by evaporation is possible. However the effect studied here is prominent with phthalic anhydride and urea, both highly involatile; and is much less significant with triethylene glycol, which as a liquid dialcohol of molecular weight 150 is certainly much more volatile than the first two.

At small particle or droplet size the efficiency of nucleation in the CNC can fall below 100% (the Kelvin effect); however this will apply equally to upstream and downstream aerosol flows and can have no effect on these measurements.

In the filter penetration work of Part I irregularities occurred below 0.05 μm, in the form of results that varied widely with challenge material, and a minimum in penetration. This is the same range for which abnormal transport losses are reported in the present work. An aerosol property which undergoes significant variation in the region of a few hundredths of a micron is mobility. Stokes law, $F = 3\pi\eta dV$, relates terminal velocity of a sphere of diameter d to the applied force, η being the medium viscosity. This must be modified at small values of d by insertion of the Cunningham correction; a form applicable at sizes in the vicinity of the mean free path in ambient air (0.066 μm) is presented in Hinds (7). Employing this, we can calculate mobility (V/F in the equation above). If one normalizes V/F at 0.3 μm as 1.0, relative mobilities at lower sizes are:

Size μm	0.3	0.2	0.1	0.05	0.03	0.02	0.01
V/F without correction	1	1.5	3	6	10	15	30
V/F with correction	1	1.80	5.52	19.0	50.4	105	429

This is a possible factor in the unusual behaviour observed in this size range; the effect of any force which impels aerosol toward a wall or filter body is magnified at small sizes. This motion is determined only by size, charge, and possibly shape, of the particles. There is in addition a specific material-dependent interaction between particles and wall or filter. The few experiments conducted with mixed aerosol materials, predominantly one material with admixture of another of very different behaviour, suggest that the presence of a material in its pure chemical form on the particle surface is important for this interaction. It should be noted that although in general apparent penetrations are greater than or approximately equal to 100%, in a few cases at 0.01 µm they are smaller (anthracene, 74%; silicone oil, 60, 51%; oxamide, 68%; DOP, 45%). Some of these refer to charged, some to neutralized aerosols. In these cases losses are greater by the downstream path and are another indication of the specificity of the chemical effect. The composition of both the aerosol and the material it contacts is involved. For HV4A filter paper this is a composite of glass and plastic fibres; in the line loss experiments the materials are steel, glass or tygon, with of course a cumulative deposit of all the aerosols used in previous testing.

Another illustration of this specificity is seen in the work of part I. Sixteen materials were used with HV4A, which remained a constant factor, and over the size range 0.1-0.3 µm the behaviour was quite uniform, with only a few aberrant results. However when this surface was modified in a few experiments by coating with silicone or paraffin, the penetration of one challenge, NaCl, increased by a factor of two to three.

In summary, while these experiments suggest the intervention of two factors, mobility and chemical interaction, no satisfactory explanation for the observations has been found. In any subsequent investigation a simple flow path should be devised, in which material, dimensions and flow rate can be systematically varied. This could be of value for an understanding of aerosol behaviour in this size range, but is outside the scope of the present programme.

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As part of a program of filter testing, monodisperse aerosols ranging in size from 0.01 to 0.30 μm were prepared from seventeen materials. With particles of a few hundredths of a micron diameter, losses in the test system lines were frequently much larger than expected, and varied greatly from one material to another. There was no obvious correlation between the magnitude of the effect and the chemical nature of the aerosol. These abnormal losses were greatly reduced or eliminated when the aerosol was passed through a neutralizer, which removed most of the particle charge in this size range. A more systematic study of the phenomenon should be made, with a system designed expressly for the purpose.

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